## PATENT ABSTRACTS OF JAPAN

(11)Publication number:

09-170068

(43) Date of publication of application: 30.06.1997

(51)Int.Cl.

C23C 14/06 C30B 29/10

C30B 29/38

// B23B 27/14

C30B 23/08

(21)Application number : 07-348309

(71)Applicant: TOSHIBA TUNGALOY CO LTD

(22)Date of filing:

18.12.1995

(72)Inventor: SUZUKI TETSUYA

**FUKANO KENJI** 

**KOBATA MAMORU** 

# (54) HIGH STRENGTH COATED MEMBER WITH CRYSTAL ORIENTATIONAL PROPERTY (57)Abstract:

PROBLEM TO BE SOLVED: To provide a high strength coated member with crystal orientational property, having a service life prolonged to a greater extent, by applying a film, having high toughness, high hardness characteristic, and resistance to wear, oxidation, thermal shock, breaking, and deposition in a wide region from low temp. region to high temp. region, and a film having peeling resistance.

SOLUTION: This member is a coated member formed by coating a base material with a hard film consisting of a monolayer or multilayer of one or more kinds among titanium- and aluminum-containing compound nitrides, compound carbonitrides, compound nitrogen oxides, and compound carbonitroxides. Moreover, an intermediate layer, consisting of a monolayer of one kind among the carbides and nitrides of the group IVa, Va, Vla metals of the periodic table and mutual solid solutions thereof or a multilayer of two or more kinds among the above substances, is interposed between the base material and the hard film. At this time, coating is performed so that the interface between the base material and the intermediate layer or/and the intermediate layer and the hard film keep a heteroepitaxial relationship.

## LEGAL STATUS

[Date of request for examination]

02.09.1999

[Date of sending the examiner's decision of

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#### **DETAILED DESCRIPTION**

[Detailed Description of the Invention] [0001]

[Field of the Invention] This invention relates to the crystal stacking tendency high intensity covering member which comes to cover the coat which comes with the compound of (TiAl) on the base material of a metal, an alloy, or a ceramic sintered compact in a hetero epitaxial relation. The high degree of hardness excellent in peeling resistance and the coat of high toughness are specifically covered on the base material of a metal, an alloy, or a ceramic sintered compact. For example, a lathe-turning tool, a milling cutter tool, a drill, the tool for cutting represented by the end mill, It is related with the crystal stacking tendency high intensity covering member optimal as cutting cutting edges, such as a slitting machine, a decision cutting edge, mold tools, such as a dice and punch, the tool for antifriction represented as corrosion-proof antifriction members, such as a nozzle, and an engineering-works construction tool represented by the bit.

[0002]

[Description of the Prior Art] Thickness covers the coat of the ceramics 20 micrometers or less on a metal, an alloy, and the base material of the ceramics, each property of a base material and a coat is pulled out effectively, and many covering members which were going to attain the longevity life are proposed. When the approach of covering a coat to this covering member is divided roughly, it has chemical vapor deposition (CVD method) and physical vapor deposition (PVD). The coat covered especially with PVD has the advantage which raises abrasion resistance among these, without degrading the reinforcement of a base material. Therefore, the present condition is that the coat of reinforcement, the drill which attaches importance to defect resistance, an end mill, and the covering cutting tool represented by the throwaway tip for milling cutters is generally covered with PVD.

[0003] In order to raise abrasion resistance from the former, covering the coat of titanium nitride is known well. However, the metal nitride which makes titanium nitride representation tends to oxidize at an elevated temperature, and has the problem that abrasion resistance deteriorates remarkably. The thing about the covering member represented by the coat of a compound (TiAl) is one of those which were proposed from the middle of the 1980s by making the problem of oxidation of this titanium nitride coat into improvement \*\*\*\*\*\*, and JP,62-56565,A, JP,6-210502,A, JP,6-210511,A, and JP,7-197235,A are in them as that typical thing.

[0004] On the other hand, crystal orientation of the coat covered on the surface of a base material is carried out, raising the adhesion of a coat is proposed, and JP,56-156767,A, JP,2-159363,A, JP,5-287322,A, JP,5-287323,A, and JP,5-295517,A are one of the typical thing.

[Problem(s) to be Solved by the Invention] It is indicated about the surface coating hard member excellent in the abrasion resistance which formed in JP,62-56565,A, JP,6-210502,A, JP,6-210511,A, and JP,7-197235,A as advanced technology about the coat of a TiAl compound the hard enveloping layer which becomes on the surface of a base in one sort of monolayers of the carbide of (TiAl), a nitride, and the carbon nitride, or two or more sorts of double layers.

[0006] Although the surface coating hard member of an indication in these official reports is a covering hard member oxidation resistance and whose abrasion resistance improved compared with the coat of Ti compound as the time of development, a mechanical property deteriorates conversely, and when it applies to a tool and the cutting tool especially used on severe conditions, it has the problem that cuttingability ability falls. That is, a surface coating hard member given in this official report While improvement in the chemical property on the front face of a coat was attained by making aluminum contain in a coat as compared with the coat of Ti compound Since crystal structure-consideration in the interface of a base material and a coat is not carried out, the peeling resistance and reinforcement of a coat are inferior, and the fracture toughness value of a surface coating hard member and defect resistance fall, When it uses as a cutting tool for high speed cutting especially, there is a problem of becoming a short life by joining with oxidation of the coat by the elevated temperature, advance of rapid wear, degradation by thermal shock nature, and \*\*-ed material.

[0007] On the other hand, it is indicated by JP,56-156767,A, JP,2-159363,A, JP,5-287322,A, JP,5-287323,A, and JP,5-295517,A as advanced technology about the coat by which crystal orientation was carried out about the covering hard member which carried out crystal orientation of the coat of Ti compound, and covered it on the surface of the base material.

[0008] Among the official reports about such crystal orientation, since the crystal stacking tendency covering hard member of an indication in front 2 official reports is not maintaining hetero epitaxial relation in the mutual interface of a base material and a coat, the crystal stacking tendency of a coat becomes weak and it has the problem that the adhesion of a coat and the reinforcement of a coat are inferior. Moreover, the crystal stacking tendency covering hard member of an indication in back 3 official reports does not have composition of the coat layer in consideration of the stress in the interface of a base material and a coat among these official reports, and since the mechanical property of the coat itself is low, there is a problem that it cannot yet be satisfied with the reinforcement of a coat, a degree of hardness, abrasion resistance, thermal resistance, and peeling resistance.

[0009] This invention is what solved the above troubles, and, specifically, aims at offer of the crystal stacking tendency high intensity covering member which becomes still longer lasting by having covered the coat with high toughness, high degree-of-hardness nature, abrasion resistance, oxidation resistance, thermal shock resistance, defect resistance, and joining-proof nature, and the coat of peeling resistance in a large field until it reaches [ from a low-temperature field ] an elevated-temperature field.

[0010]

[Problem(s) to be Solved by the Invention] The covering member which covered the hard film of the compound of (TiAl) on the base material of cemented carbide this invention persons If it was used in the elevated-temperature field, when the problem that the effectiveness is reduced will be examined to demonstrating the effectiveness which was excellent in the rate when it was used in the low-temperature field, between the hard film which becomes with the compound covered on a base material (TiAl) Intervene the interlayer who becomes with 4a and 5a of the periodic table, and the compound of 6a metals, and if these base materials, interlayers, and hard film are covered so that hetero epitaxial relation may be maintained Wear-resistant reduction does not arise until it reaches a hot field from becoming a high intensity coat and low temperature, and the peeling resistance of an interlayer and the hard film improves notably, and it comes to complete a header and this invention for a life extending. [0011] In the covering member with which the hard film with which the high intensity covering member of this invention comes on a base material by one or more sorts of monolayers in the compound nitride containing titanium and aluminum, compound carbon nitride, a compound nitric oxide, and a compound charcoal nitric oxide or the multilayer was covered The interlayer who becomes by one sort of monolayers in 4a and 5a of the periodic table, the carbide of 6a group metal, nitrides, and these mutual solid solutions or two or more sorts of multilayers is made to intervene between this base material and this hard film. It is characterized by being covered so that the interface of this base material and this interlayer or/and the interface of this interlayer and this hard film may maintain hetero epitaxial relation.

[0012]

[The mode of implementation of invention] The base material in the high intensity covering member of this invention becomes with the metal member, sintered alloy, or ceramic sintered compact which can bear the temperature heated when covering a coat. Specifically For example, the metal member represented by stainless steel, a heat-resistant alloy, high-speed steel, die steel, Ti alloy, and aluminum alloy, The ceramic sintered compact of cemented carbide, a cermet, the sintered alloy of a P / M high speed steel, an aluminum2O3 system sintered compact, an Si3N4 system sintered compact, a sialon system sintered compact, and a ZrO2 system sintered compact can be mentioned. When using as the tool for cutting, or a tool for antifriction, the base material of cemented carbide, a nitrogen content TiC system cermet, or a ceramic sintered compact is [ among these ] desirable.

[0013] Specifically, the interlayer with whom it is directly covered on this base material can mention TiC, ZrC, HfC, TaC, NbC, VC, WC, Mo2C, Cr3C2, TiN, ZrN, HfN, TaN, CrN and Ti (CN), and C (TiW), C (TiTa), CN (TiTa) and N (TiTa). These interlayers can form as one sort of monolayers, or two or more sorts of multilayers. In being hard to adjust the crystal structure of a base material interface so that it may mention later especially Between considering as a multilayer interlayer or an interlayer, and a base material, a substrate layer with high compatibility with a base material, For example, Ti, TiAl, Ti3aluminum, the metal represented by TiAl3 and W or an alloy, WC, Mo2C, Cr2N, TaN, VB2, NbB2 and TaB2, W2B5, one sort of monolayers chosen from the ceramics which becomes by the hexagonal crystal structure of MoB2 and CrB2 Or it is also desirable to form the substrate layer which becomes in two or more sorts of double layers. As for this interlayer's film thickness, specifically, it is [ that what is necessary is just the film thickness to which the hard film covered on the surface of an interlayer can maintain hetero epitaxial relation at, and can raise adhesion ] desirable 0.01-5-micrometer thickness and to become by 0.01-1-micrometer thickness especially.

[0014] Specifically (Ti, aluminum), the hard film covered by this interlayer's front face can mention N, CN (Ti, aluminum), NO (Ti, aluminum), and CNO (Ti, aluminum). As for this hard film, it is desirable to become by the film thickness it is thin by 0.5-10 micrometers, and it is desirable to become by the film thickness it is thin by 0.5-5 micrometers in the application which attaches importance to especially shock resistance.

[0015] These hard film is CyN(Tia, Alb)1-y especially, although the case where it becomes with stoichiometric composition or non-stoichiometric composition is sufficient. Ti in formula [, however a formula shows titanium, aluminum shows aluminum, a and b express the atomic ratio of Ti and aluminum, and y expresses a carbonaceous atomic ratio, respectively. Moreover, a+b=1, 0.25<=a<=0.75 It is desirable from peeling resistance to become by one or more sorts in the compound nitride which can express with] which has the relation of 0<=y<=0.95, and compound carbon nitride. When y showing the atomic ratio of the a and carbon which express the atomic ratio of Ti in an above-mentioned formula especially becomes by the hard film which is in the relation between 0.4<=a<=0.6 and 0<=y<=0.2, respectively, it is desirable from properties, such as reinforcement of the hard film and abrasion resistance, being excellent.

[0016] The high intensity covering members of this invention are that orientation of the crystal of the interlayer side by the side of an interface with a base material and/or the interlayer side by the side of an interface with the hard film is carried out, and that orientation of the crystal of the hard film surface by the side of an interface with an interlayer is carried out. Moreover, the still more desirable configuration of the high intensity covering member of this invention is that crystal orientation of the base material side which adjoins with an interlayer is carried out to the above-mentioned configuration and above-mentioned \*\* of crystal orientation.

[0017] The hetero epitaxial relation to the high intensity covering member of this invention It comes to be parallel to the crystal face (0001) of the crystal grain with which 20% or more of crystal face (1-11) of the middle class side which touches a base material by the middle class consisting of titanium nitride and/or charcoal titanium nitride constitutes a base material front face. And an interlayer's <110> crystal orientation is parallel to <11-20> crystal orientation of a base material. 20% or more of crystal face (1-11) of the field where an interlayer touches the above-mentioned hard film is parallel to the crystal face (1-11) of the crystal grain which constitutes the hard film, and becomes, and it is desirable that an

interlayer's <110> crystal orientation is especially parallel to the <110> crystal orientation of the hard film.

[0018] Furthermore, the hetero epitaxial relation to the high intensity covering member of desirable this invention Besides the crystal orientation of an above-mentioned interlayer side, the crystal structure in the front face of a base material is hexagonal, and is mainly constituted. It is that 20% or more on the front face of a base material which touches an interlayer becomes in the crystal face (0001), and it is parallel to the crystal face (1-11) of the crystal grain with which the crystal face (0001) on the front face of a base material constitutes an interlayer, and become, and <11-20> crystal orientation on the front face of a base material becomes an interlayer's <110> crystal orientation and parallel. [0019] Therefore, the base material in the high intensity covering member of this invention On the substrate layer and concrete target which become the front face of the base material of the cemented carbide contained as a principal component or the metal mentioned above, an alloy, and a ceramic sintered compact with a hexagonal thin film, hexagonal tungsten carbide For example, it is desirable to form and use WC, Mo2C, Cr2N, TaN, VB2, NbB2 and TaB2, W2B5, and one or more sorts of substrate layers of MoB2 and CrB2.

[0020] The metal member represented by the stainless steel with which the high intensity covering member of this invention is marketed from the former, a heat-resistant alloy, high-speed steel, die steel, Ti alloy, and aluminum alloy, The ceramic sintered compact of cemented carbide, a cermet, the sintered alloy of a P / M high speed steel, an aluminum2O3 system sintered compact, an Si3N4 system sintered compact, a sialon system sintered compact, and a ZrO2 system sintered compact is used as a base material. What is necessary is just to use the base material which becomes with P20-P40 which are preferably classified in the use selection criterion of the cemented carbide of JIS B4053, M20-40 and the cemented carbide quality of the material equivalent to K10-K20, and the especially desirable cemented carbide quality of the material equivalent to P30, M20, and M30. After grinding the front face of this base material if needed and performing washing processing by the supersonic wave, an organic solvent, etc., it is producible by covering a coat with the physical vapor deposition (PVD), the chemical vapor deposition (CVD method), or the plasma-CVD method currently performed from the former on a base material.

[0021] When covering a coat on a base material, according to each membraneous quality, PVD, a CVD method, or a plasma-CVD method can also be properly used including the substrate layer covered if needed. It is desirable to perform all coats by the PVD represented by the ion plating method or the sputtering method from on a production process among these, and it is desirable to carry out covering processing by the ion plating method, especially the arc ion plating method also in this.

[0022] About the case where the coat in the high intensity covering member of this invention is produced by the ion plating method, if it explains in full detail further, as a source of a metal, two kinds, titanium metal and metal aluminum, may be used independently, or a Ti-aluminum alloy may be used. Any, such as glow discharge besides arc discharge or high-frequency heating, are sufficient also as the approach of metaled ionization. Compound gas, such as ammonia containing nitrogen besides the gas for generating a nitride, i.e., nitrogen gas, may be used for the gas used by the ion plating method. When this reactant gas is introduced in a furnace, the metal or alloy as a source of a metal is ionized and negative bias is impressed to a base material, it is desirable from membranous crystal orientation becoming easy.

[0023]

[0024]

[Function] The hard film carries out the operation which raises the fracture toughness value of the whole coat, and abrasion resistance, and the high intensity covering member of this invention carries out the operation which raises adhesion in the case of the base material which carries out the operation which eases the stress to which an interlayer remains near the interface of a base material and a coat, and becomes especially by cemented carbide, and is making the property which was excellent in the whole covering member with these hetero epitaxial relation give.

[Example] After it used the cemented carbide (IISB 4053P30 fairly quality of the material) of the

configuration SNGN120408 marketed as the base material and the organic solvent washed this base material front face, it installed in the chamber of the arc discharge plasma method, and the inside of installation) and a chamber was made into the vacuum of 1.0x10-6 - 3.0x10-6Torr using the fixture which can be covered to coincidence to (flank and a rake face. Subsequently, after having held the base material at about 450 degrees C, having held the arc discharge current to about 100 A, discharging Ti metal in the N2-H2 gas ambient atmosphere and covering the middle class of TiN, held to the gas presentation and gas pressure which show the inside of a chamber in Table 1, the Ti-aluminum alloy was made to discharge for about 30 minutes, and the hard film of N (TiAl) and (TiAl) CN was compounded. The Ti-aluminum alloy as an evaporation source obtained this invention article 1-4 and the comparison article 1-3 using the target with which the presentations shown in Table 1 differ. The covering conditions of a different comparison article from the covering conditions of this invention article To have not carried out hydrogen processing in front of the coat, gas pressure, and bias voltage conditions were made into the conditions used from the former, It is having used the cemented carbide of the conventional marketing with same this invention article and presentation component for the base material of a comparison article at the list to having used the cemented carbide which made the plate crystal WC by which the field's grew up to be the base material (0001) of this invention article contain so much.

[0025] In this way, it checked that X-ray diffractometer, an electron microscope, and EDX equipment investigated each coat of the obtained this invention article 1-4 and the comparison article 1-3, and it became by the hard film of the interlayer of TiN, and a compound (Ti, aluminum). The hard film presentation component of this invention article 1-4 and the comparison article 1-3 was analyzed with X-ray diffractometer and glow-discharge-optical-emission-spectrometry equipment, and was shown in Table 2. Moreover, the scanning electron microscope investigated each coating thickness, and it wrote together the result to Table 2.

[0026] Subsequently, the equipment which scratches and is equivalent to a hardness tester performed the scratch trial of a coat using this invention article 1-4 and the comparison article 1-3, the critical load in which a coat exfoliates was searched for, and the result was written together to Table 2. Moreover, using this invention article 1-4 and the comparison article 1-3, the cutting trial by \*\*-ed material:S45C (HB [190]), cutting speed 300 m/min, delivery:0.5 mm/rev, infeed:2.0mm, cutting-time:60min, and dry type cutting trial was performed, each average flank wear width of face was measured, and the result was written together to Table 2.

[0027] About the hetero epitaxial relation between a base material, an interlayer, and the hard film When it checks using an electron diffraction method, this invention article 1-4 It comes to be parallel to the crystal face (0001) of the crystal grain with which 20% or more of crystal face (1-11) of the interlayer side which touches a base material constitutes a base material front face. And an interlayer's <110> crystal orientation is parallel to <11-20> crystal orientation of a base material. 20% or more of crystal face (1-11) of the field where an interlayer touches the hard film was parallel to the crystal face (1-11) of the crystal grain which constitutes the hard film, and became, and it checked that an interlayer's <110> crystal orientation was parallel to the <110> crystal orientation of the hard film. The crystal stacking tendency between field sides (rate) was shown in Table 3 among these contents of a check.

[Table 1]

14 **			被	夜	杂	<b>#</b>		
#		ターゲ 組成 (	ット	圧力	基件	時間	反応ガス (SCC	
15	_	Ti	Al	× 10 <sup>-</sup> 3	l '	(min)	N.	СН₄
	I	0.4	0.6	50	-400	30 -	100-150	
本	2	0.3	0.7	50	-300	30	80-100	<b>–</b> –
明	3	0.5	0.5	60	-350	80	100-150	30-60
品	4	0.4	0.6	60	-350	30	100-150	30-60
比	1	0.5	0.5	20	-100	35	100-150	
較	2	0.4	0.6	20	-100	35	100-150	
品	3	0.3	0.7	20	-100	85	120-200	<b></b>

[0029] [Table 2]

	K B	ぞ 質 膜(被膜)		切削試験 平均逃げ	スクラッ チ試験	
1		組成成分(原子比) 膜厚		面摩耗	路界荷重	
-	身	金属(原子%)	(μ <sub>m</sub> )	(mm)	(kg)	
-	1	(Tio. 55Alo. 45) N	2.7	0.15	2.5	
本発	2	(Tio. 45 Alo. 55) N	2.7	0.26	1.7	
明	3	(Tio. 35Alo. 85) Co. 1No. 9	2.3	0.34	2.0	
毌	4	(Tio. 55 Alo. 48) Co. 2 No. 8	2.4	0.34	2.0	
比	1	(Tio. 00 Alo. 40) N	3.0	0.40	試験前	
較	2	(Tio.45Alo.88) N	2.8	0.41	1.0	
	3	(Tio.4Alo.8) N	2.7	0.43	1.3	

[0030] [Table 3]

ii W		ヘテロエピタキシャル性				
料番号		基材と中間層		中間層と硬質層		
<del>"</del>	•	結晶配向	結晶配向率(%)	結晶配向	結晶配向率(%)	
本	1	有り	40	有り	70	
発	2	有り	2 0	有り	50	
明	8	有り	3 0	有り	60	
- 	4	有り	3 0	有り	<b>5</b> 0	
比	1	無し	o	無し	0	
較	2	無し	0	無し	1 0	
品品	3	無し	5	無し	1 0	

### [0031]

[Effect of the Invention] Since the peeling resistance of a coat is excellent and the coat itself has a high degree of hardness, high toughness, abrasion resistance, oxidation resistance, thermal shock resistance, defect resistance, and joining-proof nature as contrasted with the comparison article which covered the conventional N (TiAl) coat, the high intensity covering member of this invention is effective in becoming long lasting in the field which is equivalent to a high-speed-cutting field from the medium-speed cutting field in this field. Therefore, the thing for which the high intensity covering member of this invention has the outstanding effectiveness that a longevity life can be attained in a large field until it reaches [ from the low-speed cutting field which is a field of the conventional covering member ] a high-speed-cutting field, that it is effective in a longevity life being obtained especially as an end mill and a drill, and high toughness — and — high — since it is the covering member which covered the degree of hardness coat, the effectiveness which was excellent also in the deep-cuts field from the light cutting field can be demonstrated.

[Translation done.]

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## **CLAIMS**

### [Claim(s)]

[Claim 1] In the covering member with which the hard film which comes on a base material by one or more sorts of monolayers in the compound nitride containing titanium and aluminum, compound carbon nitride, a compound nitric oxide, and a compound charcoal nitric oxide or the multilayer was covered The interlayer who becomes by one sort of monolayers in 4a and 5a of the periodic table, the carbide of 6a group metal, nitrides, and these mutual solid solutions or two or more sorts of multilayers is made to intervene between this base material and this hard film. The crystal stacking tendency high intensity covering member characterized by being covered so that the interface of this base material and this interlayer or/and the interface of this interlayer and this hard film may maintain hetero epitaxial relation.

[Claim 2] The above-mentioned hard film is a crystal stacking tendency high intensity covering member according to claim 1 characterized by becoming by one or more sorts of monolayers of a compound nitride and compound carbon nitride or the multilayer containing the titanium substantially expressed with a degree type (A), and aluminum.

(Tia, Alb) CyN1-y ---- (A)

Ti in [, however the (A) type shows titanium, aluminum shows aluminum, a and b express the atomic ratio of Ti and aluminum, and y expresses a carbonaceous atomic ratio, respectively. Moreover, a+b=1, 0.25 <= a <= 0.75] which has the relation of 0 <= y <= 0.95

[Claim 3] The hard film expressed with the above-mentioned (A) formula is a crystal stacking tendency high intensity covering member according to claim 2 characterized by a and y in a formula satisfying the relation between  $0.4 \le a \le 0.6$  and  $0 \le y \le 0.2$ , respectively.

[Claim 4] The above-mentioned hard film is a crystal stacking tendency high intensity covering member according to claim 1, 2, or 3 characterized by film thickness becoming by 1-10 micrometers.

[Claim 5] The above-mentioned interlayer is claims 1, 2, and 3 characterized by film thickness becoming by 0.01-1 micrometer, or a crystal stacking tendency high intensity covering member given in four.

[Claim 6] It comes to be parallel to the crystal face (0001) of the crystal grain with which 20% or more of crystal face (1-11) of this middle class side that the above-mentioned middle class consists of titanium nitride and/or charcoal titanium nitride, and touches the above-mentioned base material constitutes this base material front face. And this interlayer's <110> crystal orientation is parallel to <11-20> crystal orientation of this base material. It comes to be parallel to the crystal face (1-11) of the crystal grain with which 20% or more of crystal face (1-11) of the field where this interlayer touches the above-mentioned hard film constitutes this hard film. And claim 1.2.3.4 characterized by this interlayer's <110> crystal orientation being parallel to the <110> crystal orientation of this hard film or the crystal stacking tendency high intensity covering member of five publications.

[Claim 7] The front face of the above-mentioned base material mainly consists of hexagonal crystals, and 20% or more on this front face of a base material that touches the above-mentioned interlayer becomes in the crystal face (0001). It comes to be parallel to the crystal face (1-11) of the crystal grain

with which the crystal face (0001) on this front face of a base material constitutes this interlayer. And the crystal stacking tendency high intensity covering member according to claim 1, 2, 3, 4, 5, or 6 characterized by <11-20> crystal orientation on this front face of a base material being parallel to this interlayer's <110> crystal orientation.

[Claim 8] The above-mentioned base material is a crystal stacking tendency high intensity covering member according to claim 1, 2, 3, 4, 5, 6, or 7 characterized by consisting of cemented carbide which contains tungsten carbide as a principal component.

[Translation done.]

#### (19) 日本国特許庁 (JP)

## (12) 公開特許公報(A)

(11)特許出願公開番号

## 特開平9-170068

(43)公開日 平成9年(1997)6月30日

(51) Int.Cl. <sup>6</sup>	識別記号 庁内整理番号	FΙ	技術表示箇所
C 2 3 C 14/06		C 2 3 C 14/06	L
C30B 29/10		C30B 29/10	
29/38		29/38	С
// B 2 3 B 27/14		B 2 3 B 27/14	Α
C30B 23/08		C30B 23/08	P
		審査請求未請求	R 請求項の数8 FD (全8頁)
(21)出顧番号	特願平7-348309	(71)出顧人 00022	1144
		東芝ク	ノンガロイ株式会社
(22)出顧日	平成7年(1995)12月18日	神奈川	県川崎市幸区堀川町580番地 ソリ
		ッドフ	<b>マクエア</b>
•		(72)発明者 鈴木	哲也
		神奈川	県川崎市幸区堀川町580番地 ソリ
		ッドフ	くクエア 東芝タンガロイ株式会社内
		(72)発明者 深野	賢治
		神奈川	県川崎市幸区堀川町580番地 ソリ
		ッドフ	くクエア 東芝タンガロイ株式会社内
•		(72)発明者 木幡	護
	•	神奈川	県川崎市幸区堀川町580番地 ソリ
		w K7	マスティア 東芝タンガロイ株式会社内

#### (54) 【発明の名称】 結晶配向性高強度被覆部材

#### (57)【要約】

【課題】 (TiA1) N被膜を被覆した従来の被覆部材は、破壊靭性値が低下すること、特に高速切削用切削工具として用いると、高温による被膜の酸化,急激な摩耗の進行,熱衝撃性による劣化および被削材との溶着により短寿命になるという課題がある。また、Ti化合物の被膜を被覆した従来の被覆部材は、膜自体が脆性なために切削用工具のような苛酷な用途では剥離またはチッピングを生じ易くて短寿命になるという課題がある。

【解決手段】基材上にチタンとアルミニウムとを含む複合窒化物、複合炭窒化物、複合窒酸化物、複合炭窒酸化物の中の1種以上の単層または多層でなる硬質膜が被覆された被覆部材において、該基材と該硬質膜との間に周期律表の4a、5a、6a族金属の炭化物、窒化物およびこれらの相互固溶体の中の1種の単層または2種以上の多層でなる中間層を介在させて、該基材と該中間層との界面または/および該中間層と該硬質膜との界面がヘテロエビタキシャル関係を保つように被覆されていることを特徴とする結晶配向性高強度被覆部材。

1

#### 【特許請求の範囲】

【請求項1】 基材上にチタンとアルミニウムとを含む複合窒化物、複合炭窒化物、複合窒酸化物、複合炭窒酸化物の中の1種以上の単層または多層でなる硬質膜が被覆された被覆部材において、該基材と該硬質膜との間に周期律表の4a、5a、6a族金属の炭化物、窒化物およびこれらの相互固溶体の中の1種の単層または2種以上の多層でなる中間層を介在させて、該基材と該中間層との界面または/および該中間層と該硬質膜との界面がヘテロエピタキシャル関係を保つように被覆されていることを特徴とする結晶配向性高強度被覆部材、

【請求項2】 上記硬質膜は、実質的に次式(A)で表 わされるチタンとアルミニウムとを含む複合窒化物、複 合炭窒化物の1種以上の単層または多層でなることを特 徴とする請求項1記載の結晶配向性高強度被覆部材。

 $(Ti_a, Al_b) C_y N_{1-y} ---- (A)$ 

[但し、(A) 式中のTiはチタン、AIはアルミニウムを示し、a、bはそれぞれTiおよびAIの原子比を、yは炭素の原子比を表す。また、a+b=1、 $0.25 \le a \le 0.75$ 、  $0 \le y \le 0.95$ の関係にある]

【請求項3】 上記(A)式で表わされる硬質膜は、式中におけるaおよびyがそれぞれ0.4≦a≦0.6および0≦y≦0.2の関係を満足することを特徴とする請求項2記載の結晶配向性高強度被覆部材。

【請求項4】 上記硬質膜は、膜厚さが1~10μmでなることを特徴とする請求項1,2または3記載の結晶配向性高強度被覆部材。

【請求項5】 上記中間層は、膜厚さが0.01~1 μ mでなることを特徴とする請求項1,2,3,または4 30 記載の結晶配向性高強度被覆部材。

【請求項6】 上記中間層は、窒化チタンおよび/または炭窒化チタンからなり、上記基材と接する該中間層面の20%以上の(1-11) 結晶面が該基材表面を構成する結晶粒の(0001) 結晶面と平行でなり、かつ該中間層の<110>結晶方向が該基材の<11-20>結晶方向と平行であり、該中間層が上記硬質膜と接する面の20%以上の(1-11) 結晶面が該硬質膜を構成する結晶粒の(1-11) 結晶面と平行でなり、かつ該中間層の<110>結晶方向が該硬質膜の<110>結 40 晶方向と平行であることを特徴とする請求項1.2.

3. 4または5記載の結晶配向性高強度被覆部材。

【請求項7】 上記基材の表面は、主として六方晶の結晶で構成され、上記中間層と接する該基材表面の20%以上が(0001)結晶面でなり、該基材表面の(0001)結晶面が該中間層を構成する結晶粒の(1-11)結晶面と平行でなり、かつ該基材表面の<11-20>結晶方向が該中間層の<110>結晶方向と平行であることを特徴とする請求項1,2,3,4,5または6記載の結晶配向性高強度被覆部材。

【請求項8】 上記基材は、炭化タングステンを主成分として含有する超硬合金からなることを特徴とする請求項1,2,3,4,5,6または7記載の結晶配向性高

【発明の詳細な説明】

[0001]

強度被覆部材。

【発明の属する技術分野】本発明は金属、合金またはセラミックス焼結体の基材上に(TiAl)の化合物でなる被膜をヘテロエピタキシャル関係に被覆してなる結晶配向性高強度被覆部材に関し、具体的には、金属、合金またはセラミックス焼結体の基材上に耐剥離性に優れた高硬度、高靭性の被膜を被覆して、例えば旋削工具、フライス工具、ドリル、エンドミルに代表される切削用工具、スリッターなどの切断刃、裁断刃とダイス、パンチなどの型工具とノズルなどの耐腐食耐摩耗部材として代表される耐摩耗用工具、ビットに代表される土木建設用工具として最適な結晶配向性高強度被覆部材に関する。【0002】

【従来の技術】金属、合金およびセラミックスの基材上 に厚さが20μm以下のセラミックスの被膜を被覆し、基材と被膜とのそれぞれの特性を有効に引き出して、長寿命を達成しようとした被覆部材が多数提案されている。この被覆部材に被膜を被覆する方法は、大別すると化学蒸着法(CVD法)と物理蒸着法(PVD法)がある。これらのうち、特にPVD法により被覆された被膜は、基材の強度を劣化させることなく耐摩耗性を高める利点がある。そのために、一般に強度、耐欠損性を重要視するドリル、エンドミル、フライス用スローアウェイチップに代表される被覆切削工具の被膜は、PVD法に 30 より被覆されているのが現状である。

【0003】従来から耐摩耗性を向上させるために窒化チタンの被膜を被覆することはよく知られている。しかしながら、窒化チタンを代表とする金属窒化物は高温で酸化されやすく、耐摩耗性が著しく劣化するという問題がある。この窒化チタン被膜の酸化の問題を改善しょうとして1980年代中頃から提案されたものに、(Ti A1)化合物の被膜に代表される被覆部材に関するものがあり、その代表的なものとして特開昭62-56565号公報、特開平6-210511号公報および特開平7-197235号公報がある。

【0004】一方、基材の表面に被覆する被膜を結晶配向させて、被膜の付着性を高めることが提案されており、その代表的なものとして特開昭56-156767号公報,特開平2-159363号公報,特開平5-287322号公報,特開平5-287323号公報および特開平5-295517号公報がある。

[0005]

【発明が解決しようとする課題】TiA1化合物の被膜 50 に関する先行技術としての特開昭62-56565号公

報,特開平6-210502号公報,特開平6-210511号公報および特開平7-197235号公報には、基体の表面に(TiAl)の炭化物,窒化物および炭窒化物のうちの1種の単層または2種以上の複層でなる硬質被覆層を形成した耐摩耗性に優れた表面被覆硬質部材について開示されている。

【0006】これらの公報に開示の表面被覆硬質部材は、開発当初の通りにTi化合物の被膜に比べて耐酸化性および耐摩耗性の向上した被覆硬質部材ではあるが、逆に機械的性質が劣化し、工具、特に苛酷な条件で用い 10られる切削工具へ適用した場合に切削性能が低下するという問題がある。つまり、同公報に記載の表面被覆硬質部材は、被膜中にAlを含有させることにより、Ti化合物の被膜に比較して被膜表面の化学的性質の向上を達成した反面、基材と被膜との界面における結晶構造的な配感がされていないことから、被膜の耐剥離性および耐欠損性が低下すること、特に高速切削用切削工具として用いると、高温による被膜の酸化、急激な摩耗の進行、熱衝撃性による劣化および被削材との溶着により短寿命にな 20るという問題がある。

【0007】一方、結晶配向された被膜に関する先行技術としての特開昭56-156767号公報、特開平2-159363号公報、特開平5-287322号公報、特開平5-287323号公報および特開平5-295517号公報には、Ti化合物の被膜を結晶配向して基材の表面に被覆した被覆硬質部材について開示されている。

【0008】これらの結晶配向に関する公報のうち、前2公報に開示の結晶配向性被覆硬質部材は、基材と被膜30との相互の界面においてヘテロエピタキシャル関係を保たせていないことから、被膜の結晶配向性が弱くなり、被膜の付着性および被膜の強度が劣るという問題がある。また、これらの公報のうち、後3公報に開示の結晶配向性被覆硬質部材は、基材と被膜との界面における応力を考慮した被膜層の構成になっていなく、かつ被膜自体の機械的性質が低いことから、被膜の強度,硬度,耐摩耗性、耐熱性および耐剥離性に未だ満足できないという問題がある。

【0009】本発明は、上述のような問題点を解決した 40 もので、具体的には、低温領域から高温領域に至るまでの広い領域において、高朝性、高硬度性、耐摩耗性、耐酸化性、耐熱衝撃性、耐欠損性、耐溶着性のある被膜および耐剥離性の被膜を被覆したことにより一層長寿命となる結晶配向性高強度被覆部材の提供を目的とする。 【0010】

【発明が解決しようとする課題】本発明者らは、超硬合 r B2の六方晶結晶構造でなるセラミックスの中から選金の基材上に(T i A l )の化合物の硬質膜を被覆した ばれた1種の単層または2種以上の複層でなる下地層を被覆部材が、低温領域で使用すると割合に優れた効果を 形成することも好ましいことである。この中間層の膜厚発揮するのに対し、高温領域で使用するとその効果が低 50 さは、中間層の表面に被覆される硬質膜がヘテロエピタ

減されるという問題を検討していたところ、基材上に被 習する(TiAl)の化合物でなる硬質膜との間に、周 期律表の4a,5a,6a金属の化合物でなる中間層を 介在し、これらの基材と中間層と硬質膜とをヘテロエピ タキシャル関係を保つように被覆すると、高強度な被膜 となること、また低温から高温の領域に至るまで耐摩耗 性の低減が生じなく、かつ中間層および硬質膜の耐剥離 性が顕著に向上し、寿命が延長するということを見出 し、本発明を完成するに至ったものである。

4

【0011】本発明の高強度被覆部材は、基材上にチタンとアルミニウムとを含む複合窒化物、複合炭窒化物、複合炭窒化物、複合炭窒酸化物、中の1種以上の単層または多層でなる硬質膜が被覆された被覆部材において、該基材と該硬質膜との間に周期律表の4a、5a、6a族金属の炭化物、窒化物およびこれらの相互固溶体の中の1種の単層または2種以上の多層でなる中間層を介在させて、該基材と該中間層との界面または/および該中間層と該硬質膜との界面がヘテロエピタキシャル関係を保つように被覆されていることを特徴とするものである。

#### [0012]

【発明の実施の態様】本発明の高強度被覆部材における基材は、被膜を被覆するときに加熱する温度に耐えることができる金属部材、焼結合金またはセラミックス焼結体でなり、具体的には、例えばステンレス鋼、耐熱合金、高速度鋼、ダイス鋼、Ti合金、Al合金に代表される金属部材、超硬合金、サーメット、粉末ハイスの焼結合金、Al2O3系焼結体、Si3N4系焼結体、サイアロン系焼結体、ZrO2系焼結体のセラミックス焼結体を挙げることができる。これらのうち、切削用工具または耐摩耗用工具として用いるときには、超硬合金、窒素含有TiC系サーメットもしくはセラミックス焼結体の基材が好ましい。

【0013】この基材上に直接被覆される中間層は、具 体的には、例えばTiC,ZrC,HfC,TaC,N bC, VC, WC, Mo<sub>2</sub>C, Cr<sub>3</sub>C<sub>2</sub>, TiN, Zr N, HfN, TaN, CrN, Ti (CN), (Ti W) C, (TiTa) C, (TiTa) CN, (TiT a) Nを挙げることができる。これらの中間層は、1種 の単層または2種以上の多層として形成することができ る。特に、後述するように、基材界面の結晶構造が調整 し難い場合には、多層の中間層とすること、または中間 層と基材との間に、基材との親和性の高い下地層、例え ばTi, TiAl, Ti3Al, TiAl3, W, に代 表される金属または合金、WC、Mo2C、Cr2N、T aN, VB2, NbB2, TaB2, W2B5, MoB2, C r B2の六方晶結晶構造でなるセラミックスの中から選 ばれた1種の単層または2種以上の複層でなる下地層を 形成することも好ましいことである。この中間層の膜厚 キシャル関係を保ち、付着性を高めることができる膜厚 さであればよく、具体的には、例えば $0.01\sim5\mu m$ 厚さ、特に $0.01\sim1\mu$ m厚さでなることが好まし

【0014】この中間層の表面に被覆される硬質膜は、 具体的には、例えば(Ti, Al) N、(Ti, Al) CN (Ti, Al) NO (Ti, Al) CNO & 挙げることができる。この硬質膜は、0.5~10 um でなる膜厚さでなることが好ましく、特に耐衝撃性を重 要視する用途においては0.5~5μmでなる膜厚さで 10 なることが好ましい。

【0015】これらの硬質膜は、化学量論組成または非 化学量論組成でなる場合でもよいが、特に、(Tia、 Alb) CyN1-y の式 [但し、式中のTiはチタン、 A 1 はアルミニウムを示し、a、bはそれぞれTiおよ びAlの原子比を、yは炭素の原子比を表す。また、a  $+b=1, 0.25 \le a \le 0.75, 0 \le y \le 0.9$ 5の関係にある]で表わせる複合窒化物および複合炭窒 化物の中の1種以上でなることが耐剥離性から好まし い。特に、上述の式中におけるTiの原子比を表わする 20 および炭素の原子比を表わすッがそれぞれ0. 4≤a≤ 0.6および0≤y≤0.2の関係にある硬質膜でなる 場合には、硬質膜の強度、耐摩耗性などの特性が優れて いることから好ましい。

【0016】本発明の高強度被覆部材は、基材との界面 側における中間層面および/または硬質膜との界面側に おける中間層面の結晶が配向されること、および中間層 との界面側における硬質膜面の結晶が配向されることで ある。また、本発明の高強度被覆部材のさらに好ましい 構成は、上述の結晶配向の構成と伴に、中間層と隣接す 30 る基材面が結晶配向されていることである。

【0017】本発明の高強度被覆部材におけるヘテロエ ピタキシャル関係は、中間層が窒化チタンおよび/また は炭窒化チタンからなり、基材と接する中間層面の20 %以上の(1-11)結晶面が基材表面を構成する結晶 粒の(0001)結晶面と平行でなり、かつ中間層の< 110>結晶方向が基材の<11-20>結晶方向と平 行であり、中間層が上記硬質膜と接する面の20%以上 の(1-11)結晶面が硬質膜を構成する結晶粒の(1 −11)結晶面と平行でなり、かつ中間層の<110> 40 結晶方向が硬質膜の<110>結晶方向と平行であるこ、 とが特に好ましい。

【0018】また、さらに好ましい本発明の高強度被覆 部材におけるヘテロエピタキシャル関係は、上述の中間 層面の結晶配向の他に、基材の表面における結晶構造が 主として六方晶で構成され、中間層と接する基材表面の 20%以上が(0001)結晶面でなり、基材表面の (0001)結晶面が中間層を構成する結晶粒の(1-11) 結晶面と平行でなり、かつ基材表面の<11-2 0>結晶方向が中間層の<110>結晶方向と平行にな 50

ることである。 【0019】したがって、本発明の高強度被覆部材にお ける基材は、六方晶炭化タングステンを主成分として含 有する超硬合金、または前述した金属、合金、セラミッ クス焼結体の基材の表面に六方晶の薄膜でなる下地層、 具体的には、例えばWC, Mo2C, Cr2N, TaN, VB2, NbB2, TaB2, W2B5, MoB2, CrB2 の1種以上の下地層を形成して用いることが好ましい。 【0020】本発明の高強度被覆部材は、従来から市販 されているステンレス鋼、耐熱合金、高速度鋼、ダイス 鋼, Ti合金, Al合金に代表される金属部材、超硬合 金, サーメット, 粉末ハイスの焼結合金、A 12O3系焼 結体、Si3N4系焼結体、サイアロン系焼結体、ZrO 2系焼結体のセラミックス焼結体を基材とし、好ましく はJIS規格B4053の超硬合金の使用選択基準の中 で分類されているP20~P40, M20~40および K10~K20相当の超硬合金材質、特に好ましくはP 30、M20、M30相当の超硬合金材質でなる基材を 用いればよい。この基材の表面を、必要に応じて研磨 し、超音波、有機溶剤などによる洗浄処理を行った後、 従来から行われている物理蒸着法 (PVD法), 化学蒸 着法(CVD法)またはプラズマCVD法により基材上 に被膜を被覆することにより作製することができる。 【0021】基材上に被膜を被覆する場合は、必要に応 じて被覆する下地層を含めて、それぞれの膜質に応じて PVD法,CVD法,またはプラズマCVD法を使い分 けることもできる。これらのうち、製造工程上から全て の被膜を、イオンプレーティング法またはスパッタリン グ法に代表されるPVD法で行うことが好ましく、この 中でもイオンプレーティング法、特にアークイオンプレ ーティング法で被覆処理することが好ましい。 【0022】本発明の高強度被覆部材における被膜をイ オンプレーティング法で作製する場合について、さらに 詳述すると、金属源としては金属チタン、金属アルミニ ウムの2種類を独立して用いてもよく、またはTi-A 1合金を使用してもよい。金属のイオン化の方法もアー ク放電の他、グロー放電または高周波加熱などのいずれ でもよい。イオンプレーティング法で使用するガスは、 窒化物を生成するためのガス、すなわち窒素ガスの他、

6

窒素を含んだアンモニアなどの化合物ガスを用いてもよ い。この反応ガスを炉内に導入し、金属源としての金属 または合金をイオン化し、基材に負のバイアスを印加す ると膜の結晶配向が容易となることから好ましい。 [0023]

【作用】本発明の高強度被覆部材は、硬質膜が被膜全体 の破壊靭性値および耐摩耗性を向上させる作用をし、か つ中間層が基材と被膜との界面近傍に残留する応力を緩 和する作用をし、特に超硬合金でなる基材の場合には付 着性を高める作用をし、これらのヘテロエピタキシャル 関係により被覆部材全体に優れた特性を付与させている

7

ものである。 【0024】

【実施例】市販されている形状SNGN120408の 超硬合金(JIS規格B4053のP30相当材質)を 基材とし、この基材表面を有機溶剤で洗浄した後、アー ク放電プラズマ法のチャンバー内に設置し、(逃げ面と すくい面へ同時に被覆できる治具を用いて設置)、チャ ンバー内を1.0×10<sup>-6</sup>~3.0×10<sup>-6</sup>Torrの 真空とした。次いで基材を約450℃、アーク放電電流 を約100Aに保持し、N2-H2ガス雰囲気中でTi金 10 属を放電してTiNの中間層を被覆した後、チャンバー 内を表1に示すガス組成およびガス圧に保持し、Ti-A 1 合金を約30分間放電させて (TiA1) Nおよび (TiAI) CNの硬質膜を合成した。蒸発源としての Ti-Al合金は、表1に示す組成の異なるターゲット を用いて本発明品1~4および比較品1~3を得た。本 発明品の被覆条件と異なる比較品の被覆条件は、被膜前 に水素処理をしなかったこと、ガス圧およびバイアス電 圧条件を従来から用いられている条件としたこと、並び に本発明品の基材には(0001)面の成長した板状晶 20 WCを多量に含有させた超硬合金を用いたのに対し、比 較品の基材には本発明品と組成成分が同一である従来の 市販の超硬合金を用いたことである。

【0025】こうして得た本発明品1~4および比較品1~3のそれぞれの被膜は、X線回折装置、電子顕微鏡およびEDX装置により調べて、TiNの中間層と(Ti、A1)化合物の硬質膜でなることを確認した。本発明品1~4および比較品1~3の硬質膜組成成分は、X

線回折装置およびグロー放電発光分析装置により解析して表2に示した。また、それぞれの被膜厚さは走査型電子顕微鏡で調べて、その結果を表2に併記した。

8

【0026】次いで、本発明品1~4および比較品1~3を用いて、引っ掻き硬さ試験機に相当する装置により被膜のスクラッチ試験を行い、被膜が剥離する臨界荷重を求めて、その結果を表2に併記した。また、本発明品1~4および比較品1~3を用いて、被削材:S45C(HB190)、切削速度300m/min、送り:0.5mm/rev、切込み:2.0mm、切削時間:60min、乾式切削試験による切削試験を行い、それぞれの平均逃げ面摩耗幅を測定し、その結果を表2に併

【0027】基材と中間層と硬質膜とのヘテロエピタキシャル関係については、電子線回折法を用いて確認したところ、本発明品1~4は、基材と接する中間層面の20%以上の(1-11)結晶面と平行でなり、かつ中間層の<110>結晶方向が基材の<11-20>結晶方向と平行であり、中間層が硬質膜と接する面の20%以上の(1-11)結晶面が硬質膜を構成する結晶粒の(1-11)結晶面と平行でなり、かつ中間層の<110>結晶方向が硬質膜の<110>結晶方向が硬質膜の<110>結晶方向が硬質膜の<110>結晶方向と平行であることを確認した。これらの確認内容のうち各界面間の結晶配向性(率)を表3に示した。

[0028]

【表1】

記した。

試 被 覆 条 件	<u> </u>
#\$ <del> </del>	
ターゲット 圧力 基体 おれ 時収	1
号 X10 <sup>-3</sup> アス (ali	N. CH.
1 0.4 0.8 50 -400 30	0 100-150
本 2 0.3 0.7 50 -300 30 発	0 80-100
明 3 0.5 0.5 60 -350 80	0 100-150 30-80
4 0.4 0.8 60 -350 30	0 100-150 30-60
1 0.5 0.5 20 -100 38	5 100-150
数 2 0.4 0.6 20 -100 35	5 100-150
品 3 0.8 0.7 2D -100 SE	5 120-200

[0029]

\* \*【表2】

1 1

1	2
	٠,

					T	
	試料	硬 質 膜(被膜)		切削試験 平均逃げ	スクラッ チ試験	
		i		, ,		
ł	番	組成成分(原子比)	膜厚	面摩耗	臨界荷重	
•	号	金属(原子%)	(µ ≡)	(mm)	(kg)	
	1	(Tio. 55 Alo. 45) N	2.7	0.15	2.5	
本発	2	(Tio.48Alo.88) N	2.7	0.28	1.7	
明	3	(Tio.35Alo.85) Co.1No.8	2.3	0.34	2.0	
强	4	(Tio. 55Alo. 45) Co. 2No. 8	2.4	0.34	2.0	
比	1	(Tio. ssAlo. 4s) N	3.0	0.40	試験前	
較	2	(Tio. 45Alo. 55) N	2.8	0.41	1.0	
딢	3	(Tio. 4Alo. 8) N	2.7	0.43	1.3	
			i		<u> </u>	

[0030]

\* \*【表3】

13		<u> </u>			1		
1	Ž.		ヘチロエピタキシ	エピタキシャル性			
料番号		基本	すと中間暦	中間層	日と製質層		
		特品配向	粧品配向率(%)	被基配向	結晶配向率(%)		
*	1	有り	4 0	有り	7 0		
発	2	有り	2 0	有り	50		
眀	3	有り	3 0	有り	6 0		
塘	4	有り	3 0	有り	5 0		
比	1	無し	0	無し	O		
較	2	無し	0	無し	1 0		
80	3	無し	5	無し	10		

#### [0031]

【発明の効果】本発明の高強度被覆部材は、従来の (T i A 1) N被膜を被覆した比較品に対比して、被膜の耐 剥離性が優れており、かつ被膜自体が高硬度、高靭性、 耐摩耗性, 耐酸化性, 耐熱衝擊性, 耐欠損性, 耐溶着性 を有していることから、この分野での中速切削領域から 高速切削領域に相当する領域において、長寿命になると いう効果がある。したがって、本発明の高強度被覆部材\* \*は、従来の被覆部材の領域である低速切削領域から高速 切削領域に至るまで広い領域で長寿命を達成できるとい う優れた効果があること、特にエンドミルおよびドリル として長寿命が得られるという効果があること、また高 **靭性および高硬度な被膜を被覆した被覆部材であること** から、軽切削領域から重切削領域においても優れた効果 を発揮できるものである。